

remarks

Reconsiderations of this application, in view of the following remarks, is respectfully requested.

I Status Of The Claims

Claim 1 has been amended to recite that the adsorbent contains porous non-activated carbon, at least one iron compound, at least one calcium compound, a portion of the carbon is present as organic nitrogen species, and that the pH of the adsorbent is basic. Claim 10 has been canceled without prejudice. New claims 29-34 have been added.

As can be seen from the specification, for example at page 12, lines 6-12, and page 13, lines 15-20, the adsorbents of the present invention are prepared by a process that does not involve an activation step. Therefore, the adsorbents of the present invention contain non-activated carbon, not activated carbon (i.e., they do not contain carbon that has been activated using, for example, chemical activators, such as zinc chloride, potassium hydroxide, or sodium hydroxide). The term "porous activated carbon" present in the specification at page 42, line 5 is a typographical error, and should read "porous carbon." The Examiner's attention is directed to the definition of "activated carbon" and "caustic-impregnated carbon" given at page 7, lines 12-15 of the specification. The recitation of "porous non-activated carbon" currently set forth in claims 1 and 29 for the adsorbents of the present invention is consistent with this definition. The term "porous non-activated carbon" does not constitute new matter.

Additional support for the amendments and new claims may be found, for example, in the specification at page 7, lines 12-15, page 10, lines 7-12, page 11, lines 9-12 and 22-24, page 14, lines 7 and 11 (Table 1), page 16, lines 14-16, page 19, lines 19-20, page 25, lines 10-11 and 24-25, Table 6 at page 26, lines 1-9, page 29, lines 13-15 and 18-20, page 32, lines 3 and 9, page 41, line 9, page 42, lines 6-7, and in original claims 1, 4, 6, 8, and 20. No new matter has been added.

Claims 1-34 are pending in this application, with claims 11-28 withdrawn from consideration by the Examiner. Claims 1-10 and 29-34 are currently at issue.

II Rejections Under 35 U.S.C. § 112

Claims 1-10 stand rejected under 35 U.S.C. § 112, first paragraph, for lack of written description. The Examiner contends that the specification does not describe the subject matter of these claims in such a way as to convey that the inventors had possession of the claimed invention at the time the application was filed. Specifically, the Examiner contends that there is insufficient disclosure of a nitrogen bearing porous carbon component, and that it is not seen how it can be stated that the lesser ingredient has incorporated nitrogen, when the product was made by heating a single material. The Examiner suggests rewriting claim 1 to indicate that the two components are different.

This rejection is respectfully traversed.

Claim 1 has been amended to clarify the composition of the adsorbent, and to recite, in part, that the adsorbent comprises 20-30% porous non-activated carbon, 70-80% inorganic matter including at least one iron compound and one calcium compound, wherein a portion of the carbon is present as organic nitrogen species.

The claimed adsorbent wherein a portion of the carbon is present as organic nitrogen species (such as amine or pyridine groups) is clearly disclosed and described in numerous sections of the specification as filed. The Examiner's attention is respectfully drawn, for example, to page 19, lines 23-25, page 21, lines 22-25, page 25, lines 15-17, page 28, lines 12-15, and page 43, lines 19-21, of the specification, and original claim 4.

Thus, the specification provides a sufficiently detailed disclosure of the claimed adsorbent so as to convey to a person skilled in the art that the applicants were in possession of the invention at the time the present application was filed. Applicants submit that claims 1-10 are definite and are fully supported by the specification as filed. Accordingly, reconsideration and withdrawal of this rejection is respectfully requested.

III Rejections Under 35 U.S.C. § 103(a)

Claims 1-10 stand rejected under 35 U.S.C. §103(a) as obvious over Khalili *et al.* (U.S. Patent No. 6,030,922, “Khalili”), in view of Matviya *et al.* (U.S. Patent No. 5,356,849, “Matviya”) for the same reasons set forth in the Office Action mailed February 5, 2004.

In the February 5, 2004 Office Action, the Examiner contended that Khalili teaches treating sludge to make an inorganic NO_x, SO_x and VOC scrubber. The Examiner conceded that Khalili does not teach the nitrogen containing material, but asserted that Matviya teaches this material as a SO_x, NO_x and organic scrubber. The Examiner concluded that it would have been obvious to use these two agents together, in order to gain their cumulative effect for gas scrubbing. With respect to claims 8-10, the Examiner conceded that the pH of the adsorbents prepared by the cited references could not be determined, but concluded that “it appears that various mixtures within the claimed scope produce the claimed pH as a result of the optimization of the amounts.”

This rejection is respectfully traversed.

The present claims are directed to an adsorbent containing 20-30% porous non-activated carbon, 70-80% inorganic matter including at least one iron compound and one calcium compound, wherein a portion of the carbon is present as organic nitrogen species and the pH of the adsorbent is basic.

The adsorbents prepared by Khalili all contain activated carbon that has been prepared by subjecting the sludge to chemical activation using zinc chloride. On the contrary, the present claims require that the carbon present in the adsorbent is a non-activated form of carbon. Khalili is silent with respect to an adsorbent that contains non-activated carbon, and does not teach or suggest a sludge derived adsorbent that contains non-activated carbon. One of ordinary skill in the art, based on the teachings of Khalili, would have had no motivation to prepare an adsorbent from sewage sludge that contains a non-activated form of carbon, and would not have had a reasonable expectation of success, based on Khalili, that such an adsorbent could be prepared.

As the Examiner concedes, Khalili is completely silent with respect to the pH of the adsorbents exemplified therein. Therefore, Khalili does not teach or suggest an adsorbent having a basic pH, as required by claim 1, much less a pH greater than 10 (as required by claim 8). Moreover, as can be seen in Table 2, at col. 6 of Khalili, the surface area of the Khalili adsorbents are all greater than 200 m²/g. Therefore, Khalili does not teach or suggest an adsorbent having a surface area of 100 - 200 m²/g, as required by claim 6. As can be seen from Table 1 of Khalili, the Khalili adsorbents do not contain iron or calcium, as required by the present claims.

Moreover, Applicants submit that the adsorbents prepared according to the Khalili process (which includes chemical activation using zinc chloride, followed by light and humidity activation stages, see abstract, col. 3, lines 47-52, and col. 4, lines 3-14) are acidic (i.e., the pH of the adsorbents prepared according to the Khalili process is less than 7) and not basic, as required by the present claims.

In support of this argument, Applicants submit herewith, as Exhibit A, a copy of a Declaration Under 37 C.F.R. § 1.132 of named inventor Dr. Teresa Bandosz ("the Bandosz Declaration"). In the Bandosz Declaration, the properties of several adsorbents prepared from sewage sludge obtained from three different sources according to the procedure set forth in Example 6 of Khalili are presented. The results are shown in Table 1 on page 6 of the Bandosz Declaration. The pH of the adsorbents prepared according to the Khalili process of Example 6 (involving chemical activation using zinc chloride, followed by light and humidity activation stages and washing with hydrochloric acid), was less than 7 (i.e., the pH of Khalili adsorbents was not basic, as required by the present claims). See paragraph 8 of the Bandosz Declaration.

Applicants also submit herewith, as Exhibit B, a copy of *Ind. Eng. Chem. Res.*, 2001, 40, pages 3502-3510. This reference describes the preparation of sewage sludge adsorbents derived from Terrene[®], whereby the adsorbents contain activated carbon (activated using a zinc chloride activation step). This is similar process to the procedure described by Khalili in Examples 1-5 and 7, and does not include the additional light or humidity activation stages described in Example 6 of Khalili.

As can be seen from Table 1 on page 3505 of Exhibit B, the pH of each of the adsorbents (SCZn-1A, SCZn-2A, SCZn-3A, and SCZn-4A) prepared by a process similar to that exemplified by Khalili in Examples 1-5 and 7 (i.e., a process that includes the zinc chloride activation and acid washing steps but no additional light or humidity activation steps) all have an acidic pH (2.47, 2.48, 3.27, and 3.29, respectively), not a basic pH as required by the present claims. Moreover, as can be seen from Table 1 of Exhibit B, the corresponding zinc chloride activated adsorbents that have not been subjected to the Khalili acid washing step (adsorbents SCZn-1, SCZn-2, SCZn-3, and SCZn-4 in Exhibit B) have significantly lower hydrogen sulfide breakthrough capacities (4, 3, 24, and 20 mg/g, respectively) than the non-activated adsorbents of the present invention prepared by pyrolysis at the same temperature (8.2, 14.9, 23.6, and 82.6 mg/g, respectively). See Table 7 at page 31 of the present specification.

Matviya is directed to the preparation of a carbonaceous char by thermal oxidation of bituminous coal followed by addition of a nitrogen containing compound (such as urea), subsequent pyrolysis, and activation using steam (see col. 4, lines 3-6).

Matviya is completely silent with respect to an adsorbent prepared from sewage sludge that contain a non-activated carbon, and does not teach or suggest an adsorbent derived from sewage sludge, wherein the carbon is non-activated. Matviya is also completely silent with respect to the pH of the adsorbents exemplified therein, and does not teach or suggest an adsorbent having a basic pH as required by claim 1, much less a pH greater than 10 (as required by claim 8). Moreover, Matviya is also completely silent with respect to the surface area of adsorbents exemplified therein. Therefore, Matviya does not teach or suggest an adsorbent having a surface area of 100 - 200 m²/g, as required by claim 6.

As set forth at page 11, lines 22-24, of the present specification, due to the basic nature of the adsorbent of the present claims, one significant advantage observed during adsorption of the acidic gas H₂S is that it is oxidized to elemental sulfur and salts thereof (thereby resulting in an environmentally inert product that can easily be disposed of) not sulfuric acid, as is observed using standard adsorbents (see page 29, lines 15-20 of the specification).

Khalili and Matviya, when taken alone or in combination, fail to teach or suggest an adsorbent containing 20-30% porous non-activated carbon, 70-80% inorganic matter including at least one iron compound and one calcium compound, wherein a portion of the carbon is present as organic nitrogen species, and the pH of the adsorbent is basic. Moreover, the cited references fail to teach or suggest that the use of such an adsorbent for the adsorption of hydrogen sulfide would result in the formation of elemental sulfur and salts thereof, and not sulfuric acid, thereby leading to an environmentally inert product that can be safely discarded.

Accordingly, the present claims would not have been obvious over the combination of Khalili and Matviya. This rejection should be withdrawn.

IV Rejections Under 35 U.S.C. § 102(b) and 103(a)

Claims 1-10 have been rejected under 35 U.S.C. §102(b) as anticipated by, or in the alternative, under 35 U.S.C. §103(a) as obvious over Lewis (U.S. Patent No. 4,122,036, "Lewis") or Khalili. The Examiner contends that both these references disclose pyrolyzing sludge, and that there is no difference between the resulting compositions and the disclosure of page 42 of the specification. In the present Office Action, the Examiner also asserts that because the references treat sludge and make a product, no differences are seen between the references and the claimed adsorbent. The Examiner concludes that the compositions of the references should be compared to the compositions of the present invention, and the claims limited to demonstrated differences.

This rejection is not believed to be well taken, and is respectfully traversed.

As conceded by the Examiner at page 2 of the Office Action, Khalili does not disclose an adsorbent that includes an organic nitrogen species, as required by the present claims. Moreover, Khalili does not teach or suggest an adsorbent containing a non-activated carbon, and does not teach or suggest the required percentage limitations of claim 1 (namely 20-30% percent non-activated carbon and 70-80% inorganic matter). With reference to the Bandosz Declaration discussed above, the Khalili adsorbents do not have a basic pH, as required by the present claims.

As stated above, the Examiner contends that there is no difference between the resulting compositions and the disclosure of page 42 of the specification. Khalili described adsorbents containing carbon in activated form, the adsorbents being formed by pyrolysis of sludge that has subjected to chemical activation using zinc chloride. As stated above, the adsorbents of the present invention are prepared solely by pyrolysis of dewatered sewage sludge, not from activated carbon that has undergone any of chemical activation, light, and/or humidity treatment steps. The adsorbents of the present invention do not contain activated carbon.

Khalili does not teach or suggest an adsorbent derived from sewage sludge that contains 20-30% porous non-activated carbon, 70-80% inorganic matter including at least one iron compound and one calcium compound, wherein a portion of the carbon is present as organic nitrogen species, and the pH of the adsorbent is basic. As can be seen from Table 1 of Khalili, the Khalili adsorbents do not contain iron or calcium, as required by the present claims. Accordingly, the claims are not anticipated by and would not have been obvious over Khalili, and the rejection should be withdrawn.

Lewis teaches a process for pyrolyzing sewage sludge to produce activated carbon, whereby recycled hot char produced during the process is added to the sewage sludge starting material (see col. 1, lines 45-46) and the material is activated using steam (see the abstract, col. 1, lines 31-32 and col. 2, lines 29-30). Lewis is completely silent respecting an adsorbent containing non-activated carbon. Lewis is also completely silent respecting the weight percent of porous carbon and inorganic matter present in the adsorbent, as well as the presence of organic nitrogen species, at least one iron compound, and at least one calcium compound, as required by claim 1. Lewis is also silent respecting the presence of catalytic oxides, the surface area of the adsorbent, and, moreover, is completely silent respecting the pH of the adsorbent.

Indeed, Lewis teaches that the quantity of char that must be recycled (which will determine the final carbon content of the activated carbon) depends on the initial sludge conditions, and operating conditions of the apparatus used (see col. 1, lines 49-52). Accordingly, the activated carbon produced by Lewis will not necessarily have 20-30% porous non-activated carbon, 70-80%

inorganic matter including at least one iron compound and one calcium compound, wherein a portion of the carbon is present as organic nitrogen species, and have a basic pH, as required by the present claims.

Anticipation requires that each and every element of the rejected claim be disclosed in a single prior art reference. See, M.P.E.P. § 2131. Every element of the claimed invention must be literally present, arranged as in the claim. *Perkin Elmer Corp. v. Computervision Corp.*, 732 F.2d 888, 894, 221 USPQ 669, 673 (Fed. Cir. 1984). A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference." *Verdegaal Bros. v. Union Oil Co. of California*, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987).

"To establish inherency, the extrinsic evidence 'must make clear that the missing descriptive matter is **necessarily** present in the thing described in the reference, and that it would be so recognized by persons of ordinary skill." *In re Robertson*, 169 F.3d 743, 745, 49 USPQ2d 1949, 1950-51 (Fed. Cir. 1999); *Continental Can Co. U.S.A. v. Monsanto Co.*, 948 F.2d 1264, 20 USPQ2d 1746 (Fed. Cir. 1991). See also, *Finnigan Corp. v. ITC*, 51 USPQ2d 1001, 1009 (Fed. Cir. 1999). "Inherency, however, may not be established by probabilities or possibilities. The mere fact that a certain thing may result from a given set of circumstances is not sufficient." *Finnigan Corp.* at 1009 (quoting *In re Oelrich*, 666 F.2d 578, 581, 212 USPQ 323, 326 (CCPA 1981)).

Consequently, when rejecting claims as being inherently anticipated by a cited prior art reference, the Examiner **must** show that the claimed limitations **necessarily** flow from the teachings of the cited prior art. See, M.P.E.P. § 2112; quoting *Ex parte Levy*, 17 USPQ2d 1461, 1464 (Bd. Pat. App & Inter. 1990) ("In relying upon the theory of inherency, the examiner must provide a basis in fact and/or technical reasoning to reasonably support the determination that the allegedly inherent characteristic **necessarily** flows from the teachings of the applied prior art."). Merely suggesting that it is possible or even probable that something described in the prior art is inherently the same as a claimed invention is not sufficient for anticipation under 35 U.S.C. § 102. As explained above, there is no evidence demonstrating that the adsorbents described in Lewis

would necessarily contain 20-30% porous carbon, 70-80% inorganic matter including at least one iron compound and one calcium compound, wherein a portion of the carbon is present as organic nitrogen species, and have a basic pH, as required by the present claims.

For a reference to inherently anticipate, the claimed construction must necessarily be present in the reference. *In re Robertson*, 169 F.3d 743, 745, 49 USPQ2d 1949, 1950-51 (Fed. Cir. 1999). As pointed out above, that is not the case here. Accordingly, Lewis does not anticipate the present claims.

As stated above, due to the basic nature of the adsorbent of the present claims, one significant advantage observed during adsorption of the acidic gas H₂S is that it is oxidized to elemental sulfur and salts thereof (thereby resulting in an environmentally inert product that can easily be disposed of) not sulfuric acid, as is observed using standard adsorbents (see page 11, lines 22-24, and page 29, lines 15-20, of the specification).

Lewis fails to teach or suggest an adsorbent containing 20-30% porous non-activated carbon, 70-80% inorganic matter including at least one iron compound and one calcium compound, wherein a portion of the carbon is present as organic nitrogen species and the pH of the adsorbent is basic. Moreover, Lewis fails to teach or suggest that the use of such an adsorbent for the adsorption of hydrogen sulfide would result in the formation of elemental sulfur and salts thereof, not sulfuric acid, thereby leading to an environmentally inert product that can be safely discarded. Therefore, the present claims would not have been obvious over Lewis.

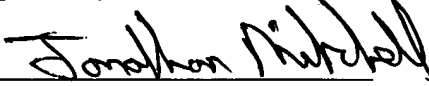
Applicants submit that the present claims are not anticipated by and would not have been obvious over Lewis or Khalili, either taken alone or in combination, and respectfully request that the rejections be withdrawn.

In view of the above arguments, the pending claims in this application are believed to be in condition for allowance. Accordingly, the Examiner is respectfully requested to enter this Amendment, and to pass this application to issue.

If there are any other issues remaining which the Examiner believes could be resolved through either a Supplemental Response or an Examiner's Amendment, the Examiner is respectfully requested to contact the undersigned at the telephone number indicated below before an Office Action is mailed.

Dated: November 19, 2004

Respectfully submitted,

By 

Jonathan P. Mitchell, Ph.D.

Registration No.: 50,239

DARBY & DARBY P.C.

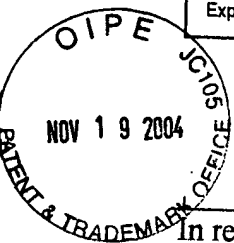
P.O. Box 5257

New York, New York 10150-5257

(212) 527-7700

(212) 753-6237 (Fax)

Attorneys/Agents For Applicant



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Docket No.: 02830/100H701-US1
(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Teresa J. Bandosz *et al.*

Confirmation No. 2434

Application No.: 09/997,398

Art Unit: 1754

Filed: November 29, 2001

Examiner: S. L. Hendrickson

For: PROCESS TO PREPARE ADSORBENTS
FROM ORGANIC FERTILIZER AND THEIR
APPLICATIONS FOR REMOVAL OF ACIDIC
GASES FROM WET AIR STREAMS

DECLARATION OF TERESA J. BANDOSZ, Ph.D., D.Sc. UNDER 37 C.F.R. § 1.132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

I, Teresa J. Bandosz, Ph.D., D.Sc. hereby declare as follows:

1. I am a citizen of the United States and I am more than 21 years of age. I have a B.Sc. conferred in 1984 from the Department of Processing of Coal in Power Engineering Chemistry of the University of Mining and Metallurgy, Krakow, Poland. I have a Ph.D. in chemical engineering, conferred in June 1989 from the Department of Chemical Engineering and Technology, Technical University of Krakow, Poland. I have a D.Sc. in physical/analytical chemistry, conferred in June 1998 from the M. Curie-Sklodowska University, Lublin, Poland. From January 1991 to July 1996 I worked as a Post-Doctoral Research Assistant in the Department of Chemical Engineering and Materials Science at the University of Syracuse, New York. From August 1984 to January 1991, I was an Assistant Professor at the University of Mining and

Metallurgy and the Institute of Energochemistry of Coal and Physical Chemistry of Sorbents, both of which are located in Krakow, Poland. Since August 1996, I have been employed in the Chemistry Department of the City College at the City University of New York, the assignee of the present patent application (as a Grant Assistant Professor from August 1996 to August 1997, as an Assistant Professor from September 1997 to 2000, and as an Associate Professor from January 2002 to the present). From 1984 to the present, I have been actively involved in the development of processes for manufacturing adsorbents, including those derived from sewage sludge. I am author of 136 journal articles covering the preparation and properties of adsorbents, and an inventor on 3 U.S. patents applications covering carbon based adsorbents. I have also made 74 presentations of my research work on adsorbents at International conferences in the U.S. Europe, and Asia. A copy of my *curriculum vitae* is attached at Exhibit 1.

2. I am a co-inventor of the subject application, and have reviewed the specification and the claims as amended in the Amendment accompanying this Declaration.

3. I understand that claims 1-10 of the present application cover an adsorbent containing 20-30% porous non-activated carbon, 70-80% inorganic matter including at least one iron compound and at least one calcium compound, wherein a portion of the carbon is present as organic nitrogen species and the pH of the adsorbent is basic; that claims 11-28 have been withdrawn from consideration by the Examiner; and that claims 29-33 cover an adsorbent containing 20-30% porous non-activated carbon, 70-80% inorganic matter including at least one iron compound and at least one calcium compound, wherein a portion of the carbon is present as

organic nitrogen species, the pH of the adsorbent is basic, and the adsorbent reacts with hydrogen sulfide to produce elemental sulfur and salts thereof.

4. I understand that claims 1-10 of the present application have been rejected by the United States Patent and Trademark Office as anticipated by Khalili's U.S. Patent No. 6,030,922 ("Khalili"), or as obvious over Khalili when taken alone or in combination with Matviya's U.S. Patent No. 5,356,849. I am familiar with the Khalili and Matviya patents.

5. On September 13, 2004, I was asked by patent attorneys of the City University of New York to make sewage sludge derived adsorbents according to the procedure described in Khalili, and to measure the pH of the resulting adsorbent. I was also asked to measure the pH of each of these adsorbents after exhaustive adsorption of hydrogen sulfide. I planned and organized these experiments, which were completed on October 21, 2004 under my supervision at the laboratories of the City University of New York. The results of these tests are described in paragraphs 8-11 below.

6. Sewage sludge used as a starting material in the procedure described in Khalili was obtained from the following three sources:

(1) Philadelphia Water Department, Biosolids Recycling Center, 7800 Penrose Ferry Road, Philadelphia, Pennsylvania, 19153;

(2) Veolia Water North America Operating services, LLC, PO Box 9856, East 12th Street & Hay Road, Wilmington, Delaware, 19809; and

(3) City of Baltimore, Department of Public Works, Bureau of Water and Wastewater, Environmental Services Division, Water and Wastewater Facilities, Back River Wastewater Treatment Plant, 8201 Eastern Boulevard, Baltimore, Maryland 21224.

7. Khalili is directed to sewage sludge derived adsorbents, prepared by a process that involves zinc chloride activation, followed by treatment with hydrochloric acid. This procedure is given in Example 6 of Khalili. Therefore, sewage sludge-derived adsorbents were prepared according to the procedure disclosed in Example 6 of Khalili. Specifically, 50 g of crushed sewage sludge material was mixed with 30.1 ml of a saturated solution of zinc chloride (50 g zinc chloride in 43 ml of water, 5 moles/liter, density = 2.12 g/ml). The ratio of sewage sludge to zinc chloride was 1:1. The mixture was stirred and heated continuously for 7 hours at 85°C and then dried overnight at 108 °C. The resulting product was exposed to light at an average intensity of 50 microwatts and a relative humidity of 60-70% for a duration of 22 hours. The sample was then dried at 108 °C for 26 hours. Subsequently, the sample was crushed and sieved, with the fraction of particles sized between 1 mm and 3 mm being selected for pyrolysis. The sample was pyrolyzed at 800 °C under a constant nitrogen atmosphere. The heating rate was 20 °C/minute, and the hold time at 800 °C was 2 hours. The sample was then cooled under a nitrogen flow, rinsed with 1M hydrochloric acid and then water, and the resulting adsorbent dried at 120 °C for 6 hours. The adsorbent prepared from sewage sludge starting material (1) was identified as SAMPLE 1. The adsorbent prepared from sewage sludge starting material (2) was identified as SAMPLE 2. The adsorbent prepared from sewage sludge starting material (3) was identified as SAMPLE 3.

8. The pH of each sample was determined using the procedure set forth at page 18, lines 7-10 of the specification. Specifically, a 0.4 g sample of the dry adsorbent was added to 20 ml of water and the suspension stirred overnight to reach equilibrium. The sample was filtered and the pH of the solution measured using an Accumet Basic pH meter. SAMPLE 1 had an initial pH of 6.3, SAMPLE 2 had an initial pH of 5.63, and SAMPLE 3 had an initial pH of 5.25. See Table 1 at paragraph 10 below.

9. Adsorbent SAMPLES 1, 2, and 3 were then subjected to exhaustive adsorption of hydrogen sulfide (H_2S) using the procedure set forth in the specification at page 18, line 22 to page 19, line 4. Specifically, the adsorbent sample was packed into a column (length 60 mm, diameter 9 mm, bed volume 6 cm^3) and pre-humidified with moist air (relative humidity 80% at 25 °C) for one hour. Moist air (relative humidity 80% at 25 °C) containing 0.3% (3000 ppm) hydrogen sulfide was then passed through the column of adsorbent at a rate of 0.5 l/min. The elution of hydrogen sulfide was monitored using an Interscan LD-17 hydrogen sulfide continuous monitor system interfaced with a computer data acquisition program. The test was stopped at the breakthrough concentration of 500 ppm. The pH of the adsorbent after exhaustive adsorption of hydrogen sulfide was then determined. SAMPLE 1 had a final pH of 6.19, SAMPLE 2 had a final pH of 2.8, and SAMPLE 3 had a final pH of 2.08.

10. The pH of SAMPLES 1-3 before and after exhaustive hydrogen sulfide adsorption are given in TABLE 1. For comparison purposes, corresponding data for SAMPLES SC-3 and SC-4 (adsorbents of the present invention, prepared by pyrolysis at 800 °C and 950 °C, respectively) is also presented in Table 1.

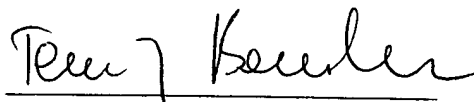
TABLE 1

Adsorbent Sample	pH before H₂S adsorption	pH after H₂S adsorption
SAMPLE 1	6.3	6.19
SAMPLE 2	5.63	2.8
SAMPLE 3	5.25	2.08
SAMPLE SC-3	11.29	8.8
SAMPLE SC-4	10.84	9.9

11. Adsorbent SAMPLES 1-3, prepared according to the process disclosed in Example 6 of Khalili, have an initial pH less than 7 (i.e. the adsorbents are acidic). After exhaustive adsorption of hydrogen sulfide, the pH of adsorbent SAMPLES 1-3 is also acidic. In my opinion, the reduction in pH of the adsorbent SAMPLES 2 and 3 after exhaustive hydrogen sulfide adsorption is due to the formation of sulfuric acid. On the contrary, the adsorbents of the present invention have a pH that is basic. The pH of the adsorbents of the present invention remains basic after exhaustive adsorption of hydrogen sulfide.

12. I further declare that all statements made herein are based on information and belief and are believed to be true and that these statements were made with the knowledge that willful false statements made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code and that such willful false statement may jeopardize the validity of the application or any patent issuing thereon.

Date 11/16/04



Teresa J. Bandosz, Ph.D., D.Sc.

H₂S Adsorption/Oxidation on Adsorbents Obtained from Pyrolysis of Sewage-Sludge-Derived Fertilizer Using Zinc Chloride Activation

Andrey Bagreev,^{†,‡} David C. Locke,[§] and Teresa J. Bandosz^{*,†}

Department of Chemistry and The International Center for Environmental Resources and Development, City College of New York, City University of New York, New York, New York 10031, and Department of Chemistry, Queens College, City University of New York, Flushing, New York 11367

Sewage-sludge-derived fertilizer, Terrene, was used as a precursor of adsorbents tested for removal of hydrogen sulfide from moist air. The adsorbents were obtained by pyrolysis of zinc chloride-impregnated granular fertilizer at 400, 600, 800, and 950 °C in a nitrogen atmosphere. Subsamples of the materials obtained were washed with hydrochloric acid to remove the excess zinc chloride and other soluble chlorides. This treatment results in a significant increase in the sample porosity. The highest H₂S removal capacity was obtained for the sample carbonized at 600 °C. This results from release of zinc chloride vapor during heat treatment, which acts to form micropores in the carbonaceous deposit. When inorganic oxides and salts are present in sufficient quantities, hydrogen sulfide undergoes surface reactions and is left on the surface in the form of nonvolatile sulfides and sulfates. The pores are gradually filled as the surface reaction proceeds. If catalytic metal oxides are removed, hydrogen sulfide is oxidized predominantly to sulfuric acid through the catalytic effect of the adsorption sites in the small micropores of the carbonaceous deposit. The removal of H₂S occurs until all pore entrances are blocked with the oxidation product.

Introduction

Growing environmental awareness directs the attention of researchers toward new and alternative methods of waste reduction and waste minimization.¹ One of the waste materials produced in abundant quantity in wastewater treatment plants is municipal sewage sludge, referred to as biosolids. Biosolids consist of organic material, mainly dead bacterial cells; inorganic components in the form of various oxides and salts (aluminum, silicon, calcium, iron, etc.); and heavy metal contaminants such as lead and copper from plumbing. Other metals present include nickel and zinc.^{2–4}

Biosolids disposal methods include landfilling, incineration, and road paving. Biosolids can also be converted into adsorbents for wastewater pollutants or applied to soil as fertilizer.⁵ Recently, the agricultural application of biosolids has been a topic of major controversy² because of the presence of heavy metals considered toxic to the environment. Metals such as lead, copper, and cadmium can accumulate in soils and, after extensive application of biosolids-derived fertilizer, can leave the soil permanently contaminated and not safe for the growing of food crops. It is interesting that U.S. EPA standards for heavy metals in fertilizers are up to 100 times higher than the limits allowed in any other country.² This could significantly increase the risk of permanent contamination and damage to ecological systems.

Continuous increases in the quantity of sludge produced, especially in Europe, call out for efficient and

environmentally friendly approaches to its utilization. One of these is conversion of sewage sludge into adsorbents. Since the 1970s, several patents have been issued proposing carbonization of sewage sludge^{6–10} and application of the carbonized material to the removal of organics in the final stages of water cleaning⁸ and to the removal of chlorinated organics.⁹ Sewage-sludge-derived adsorbents have been also tested for their ability to remove acidic gases such as sulfur dioxide and hydrogen sulfide from air streams.^{11,12} However, the reported capacity was not promising for their effective application.

Our recent results showed that materials obtained by pyrolysis of sewage-sludge-derived organic fertilizer, Terrene, perform very well as adsorbents of hydrogen sulfide.⁴ Their removal capacity is comparable to the capacity of coconut-shell-based activated carbon, which is considered as an alternative material to replace the caustic-impregnated carbons used extensively for odor control in sewage treatment plants.^{13,14}

The objective of this paper is to demonstrate the H₂S removal performance of materials obtained by chemical activation of Terrene using zinc chloride. The use of zinc chloride for chemical activation was proposed by Kemmer et al.⁹ and applied by Chiang and You¹⁵ and Lu and co-workers.^{11,12} Pyrolysis in the presence of a chemical agent significantly increases the microporosity, as well as the yield of carbonaceous phase.^{9,11,12} These results emphasize the importance of the role of pore volume and the chemistry of the adsorbent in the process of hydrogen sulfide removal. The data obtained support our hypothesis concerning the significance of the presence of catalytic metals and their surface dispersion to the adsorption and conversion of hydrogen sulfide to water-soluble species, which makes adsorbent regeneration feasible using simple methods, such as water washing.

* To whom correspondence should be addressed. E-mail: tbandosz@scisun.sci.cny.cuny.edu. Tel: (212) 650-6017. Fax: (212) 650-6107.

[†] City College of New York.

[‡] Permanent address: Institute for Sorption and Problems of Endoecology, Ukraine.

[§] Queens College.

Experimental Section

Materials. Terrene was obtained from the New York Organic Fertilizer Company (Bronx, New York) in the form of 3-mm-diameter granules with approximately 5% water content. The detailed chemical composition is presented elsewhere.³ This material contains approximately 35% inorganic matter, mainly in the form of iron, alumina, and silica oxides and carbonates, and approximately 60% organic matter.¹⁶ The adsorbents studied were prepared by pyrolysis of 10 g of Terrene impregnated with 2 mL of saturated zinc chloride solution (fertilizer weight/ZnCl₂ weight = 1:0.31, 23.7% ZnCl₂) at temperatures between 400 and 950 °C in a nitrogen atmosphere in a fixed bed (horizontal furnace). For all samples, the heating rate was 10 °C/min, with a soaking time of 1 h at the maximum pyrolysis temperature. The samples are referred to as SCZn-1, SCZn-2, SCZn-3, and SCZn-4, where 1, 2, 3, and 4 represent the pyrolysis temperatures of 400, 600, 800, and 950 °C, respectively. After pyrolysis, the granules were preserved with a size of 2 mm. To determine the effect of removal of metals such as iron, zinc, and copper, 10 mL of each sample was treated with 30 mL of 18% hydrochloric acid for 4 days. Then, 1 mL of 30% H₂O₂ was added, and the sample was washed with distilled water to remove the excess acid and oxidant. The samples obtained in this way are designated as SCZn-1A, SCZn-2A, SCZn-3A, and SCZn-4A.

A subsample of SCZn-2 was washed with distilled water in a Soxhlet extractor for 160 min (3 cycles per hour) to remove the soluble excess zinc chloride. This sample is referred to as SCZn-2W.

The prepared materials were studied as hydrogen sulfide adsorbents in the dynamic tests described below. After exhaustion of its adsorption capacity, each sample is identified by addition of the letter E to its designation.

Methods. H₂S Breakthrough Capacity. The dynamic tests were carried out at room temperature to evaluate the capacity of the sorbents for H₂S removal. Adsorbent samples were packed into a column (length of 60 mm, diameter of 9 mm, bed volume of 6 cm³) and prehumidified with moist air (relative humidity of 80% at 25 °C) for an hour. The amount of adsorbed water was estimated from the increase in the sample weight. Moist air (relative humidity of 80% at 25 °C) containing 0.3% (3000 ppm) H₂S was then passed through the column of adsorbent at 0.5 L/min. The elution of H₂S was monitored using an LD-17 H₂S continuous monitor system (Interscan) interfaced with a computer data acquisition program. The test was stopped arbitrarily at the breakthrough concentration of 500 ppm. The adsorption capacity of each sorbent in terms of grams of H₂S per gram of carbon was calculated by integration of the area above the breakthrough curve and from the H₂S concentration in the inlet gas, the flow rate, the breakthrough time, and the mass of sorbent.

Thermal Analysis. Thermal analysis was carried out using a TA Instruments thermal analyzer (TA Instruments, New Castle, DE). The instrument settings were a heating rate of 10 °C/min in nitrogen atmosphere at a flow rate of 100 mL/min.

pH. A 0.4-g sample of dry adsorbent was added to 20 mL of deionized water, and the suspension was stirred overnight to reach equilibrium. The sample was filtered, and the pH of the solution was measured using an Accumet Basic pH meter (Fisher Scientific, Springfield, NJ).

Table 1. Yield (%), Carbon Content (%), pH, and H₂S Breakthrough Capacities of Adsorbent Materials Studied

sample	yield of adsorbent	carbon content	pH	H ₂ S breakthrough capacity (mg/g)
SCZn-1	60.8	13.27	5.42	4
SCZn-1E	—	—	5.08	—
SCZn-2	50.0	19.37	5.46	3
SCZn-2E	—	—	5.39	—
SCZn-3	40.0	25.90	7.05	24
SCZn-3E	—	—	6.22	—
SCZn-4	36.4	28.47	9.10	20
SCZn-4E	—	—	6.90	—
SCZn-1A	29.9	42.05	2.47	15
SCZn-1AE	—	—	2.40	—
SCZn-2A	31.2	43.76	2.48	52
SCZn-2AE	—	—	1.81	—
SCZn-3A	27.2	43.13	3.27	39
SCZn-3AE	—	—	1.83	—
SCZn-4A	26.5	37.61	3.29	41
SCZn-4AE	—	—	1.85	—
SCZn-2W	44.3	19.61	5.95	8
SCZn-2WE	—	—	5.98	—

Determination of Zinc. The quantities of zinc removed from sludge-derived adsorbents during acid treatment and Soxhlet washing were determined using a Computrace 716 polarograph (Metrohm, Brinkmann Instruments, Westbury, NY) in the differential pulse mode. In all cases, the standard addition method was applied using a 0.2-mL sample and 10 mL of supporting electrolyte (0.05 M NH₃/NH₄Cl buffer solution). The differential pulse polarography peak for zinc was at -1.07 V vs SCE.

Nitrogen Adsorption. Nitrogen adsorption isotherms were measured using an ASAP 2010 analyzer (Micromeritics, Norcross, GA) at -196 °C. Before these experiments, the samples were degassed at 120 °C to a constant 10⁻⁵ Torr vacuum. Usually, the degassing temperature should be kept higher than necessary for the removal of physically adsorbed water, and 120 °C is the temperature used when one does not want to alter surface chemistry of materials (decomposition of surface groups, dehydroxylation, etc.). Because, in our case, a high vacuum was applied (10⁻⁵ Torr), we assume that all condensed adsorbate was removed from the pore system. On the other hand, we did not want to remove oxidation products, which are bound to the surface. A higher outgassing temperature would have resulted in the removal of elemental sulfur. The nitrogen adsorption isotherms were used to calculate the specific surface areas *S*_{BET} and *S*_{DFT}, the micropore volume *V*_{mic}, the total pore volume *V*_t, and the pore size distribution. The pore volumes were calculated using density functional theory.^{17,18}

Results and Discussion

Table 1 summarizes the yields of the preparation process (pyrolysis and pyrolysis combined with acid washing), the contents of the organic (carbonaceous) phase, and the pH values for the materials obtained. The yields of the material decreases with increasing temperature of carbonization as a result of the volatilization of organic compounds and the decomposition and dehydroxylation of inorganic components.³ On the other hand, the carbon content increases with increasing carbonization temperature. As expected for zinc chloride activation, the carbon content is higher than that obtained through carbonization with no dehydrating agent.¹⁹⁻²² Further, the yield of the carbonaceous phase

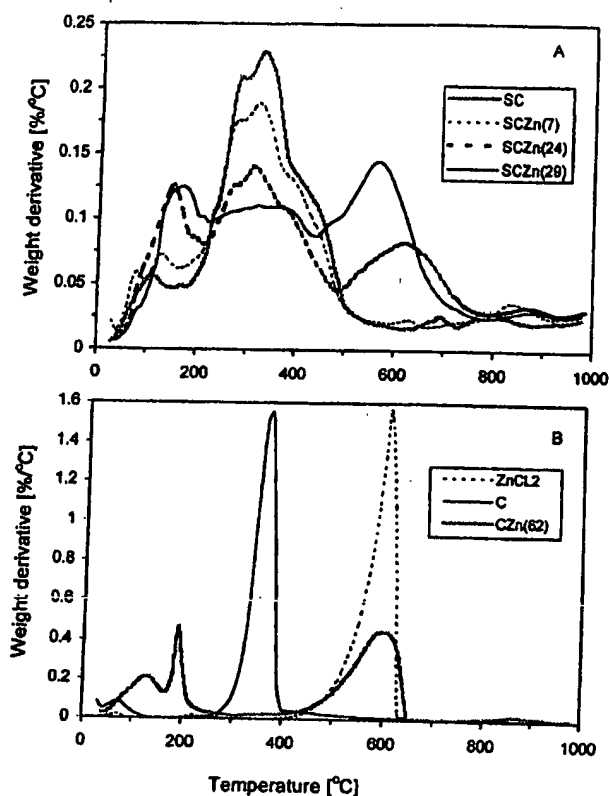


Figure 1. (A) Comparison of DTG curves in nitrogen for Terrene (SC) and Terrene impregnated with zinc chloride (SCZn). The numbers following Zn represent the percentage of ZnCl₂ in the sample. (B) DTG curves in nitrogen for pure ZnCl₂ (ZnCl₂), cellulose (C), and cellulose impregnated with zinc chloride (CZn(62)).

is higher than that obtained by simple pyrolysis of sewage sludge.³ According to the mechanism of chemical activation of organic precursors with zinc chloride, during pyrolysis, dehydration of the cellulosic material, which results in charring and aromatization of carbon skeleton, occurs. Zinc chloride acts as a dehydrating agent and inhibitor of the formation of tar.^{19–22} After the samples were washed with hydrochloric acid, the relative carbon contents increased significantly. This increase is larger for the low-temperature carbonized materials, indicating differences in the content of inorganic species and/or in the susceptibility of these species to acid treatment. The results suggest that the samples treated at high temperature have a smaller content of inorganic phase than those obtained at low temperature and are more resistant toward reaction with HCl. The removal of significant amounts of the inorganic phase as a result of acid treatment is shown in Table 1; there is an approximately 50% decrease in the total yield of the material compared to the initial samples. The results shown in Table 1 also demonstrate that sorbents obtained by pyrolysis at temperatures lower than 800 °C are acidic. The acid/base chemistry of the solids changes toward basic when pyrolysis combined with activation is carried out at 800 and 950 °C, suggesting significant changes in chemical composition.

Differences in the mechanism of Terrene carbonization with and without zinc chloride are illustrated in Figure 1A. Peaks on the DTG curves in nitrogen represent weight losses over various temperature ranges. For pyrolysis of Terrene without any activation agent,

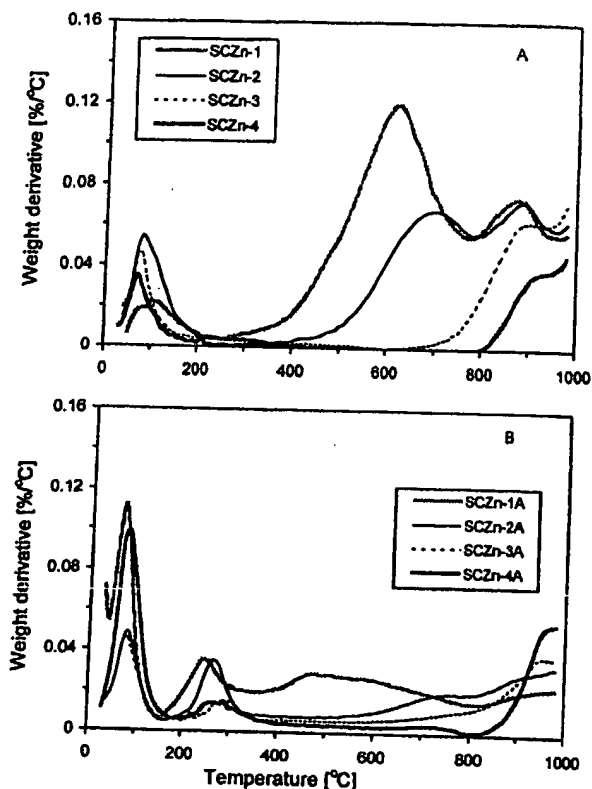


Figure 2. Comparison of DTG curves in nitrogen for sludge-derived materials (A) before and (B) after acid washing. The adsorbents initially contained 24% zinc chloride. The numbers 1–4 following Zn represent pyrolysis temperatures of 400, 600, 800, and 950 °C, respectively.

a broad peak is found between 200 and 450 °C, which represents the removal of volatile matter consisting of organic compounds and water from the decomposition of inorganic species. When zinc chloride is added, the curve shows completely different features with three broad peaks. To determine the effect of zinc chloride, different amounts of this compound were added to the fertilizer. As shown in Figure 1A, as the ZnCl₂ content was increased from 7 to 29%, an increase in the intensities of the first and the third peaks on the DTG curves resulted. Judging from these results, we assigned the third peak, centered at about 600 °C, to the evaporation of excess zinc chloride. To support this hypothesis, TG analysis of crystalline ZnCl₂ was carried out; the resulting DTG curve in nitrogen is included in Figure 1B. In addition to small weight losses at about 200 °C due to dehydration (1.3%), the sharp peak at 600 °C is present, representing removal of zinc chloride by evaporation (96% weight loss). This evaporation might play a crucial role in the process of pore formation. It is likely that zinc chloride molecules released in this process act as pore-forming agents in the carbon deposit. A similar phenomenon was observed with carbons obtained within the interlayer space of pillared clays, where released water from dehydroxylation played an important role in the pore-development process.²³ If this hypothesis is correct, then materials pyrolyzed at temperatures higher than 600 °C should become more basic as a result of the removal of zinc chloride, as observed (Table 1), and should have the highest surface areas and pore volumes. The second peak on the DTG curves in nitrogen of the Terrene/ZnCl₂ mixture, at the same

Table 2. Structural Parameters Calculated from Nitrogen Adsorption Isotherms, Decreases of Micropore (ΔV_{mic}) and Total Pore Volumes (ΔV_t), and Estimated Volumes of Adsorbed Sulfur^a

sample	S_{BET} (m ² /g)	S_{DFT} (m ² /g)	$V_{mic}(DFT)$ (cm ³ /g)	$V_t(DFT)$ (cm ³ /g)	V_{mic}/V_t	ΔV_{mic} (cm ³ /g)	ΔV_t (cm ³ /g)	V_{sulf} (cm ³ /g)
SCZn-1	4	2	0.001	0.019	0.052	—	—	—
SCZn-1E	—	—	—	—	—	—	—	—
SCZn-2	54	51	0.017	0.031	0.548	—	—	—
SCZn-2E	49	46	0.016	0.026	0.615	0.001	0.005	0.002
SCZn-3	200	219	0.068	0.094	0.723	—	—	—
SCZn-3E	173	161	0.056	0.094	0.596	0.012	0.000	0.012
SCZn-4	225	200	0.070	0.132	0.530	—	—	—
SCZn-4E	159	166	0.055	0.096	0.573	0.015	0.036	0.010
SCZn-1A	98	98	0.030	0.061	0.492	—	—	—
SCZn-1AE	58	55	0.019	0.052	0.365	0.011	0.009	0.007
SCZn-2A	397	375	0.122	0.262	0.465	—	—	—
SCZn-2AE	194	187	0.062	0.134	0.462	0.060	0.128	0.026
SCZn-3A	311	344	0.108	0.189	0.571	—	—	—
SCZn-3AE	107	103	0.028	0.099	0.282	0.080	0.090	0.020
SCZn-4A	297	279	0.090	0.195	0.461	—	—	—
SCZn-4AE	86	82	0.021	0.077	0.272	0.069	0.118	0.020
SCZn-2W	233	253	0.091	0.127	0.716	—	—	—
SCZn-2WE	189	184	0.063	0.102	0.618	0.028	0.025	0.004

^a Sulfur density assumed to be 2 g/cm³.

position as for pyrolysis of Terrene, is related to the removal of volatile organic compounds created during carbonization.

Figure 1B shows the effect of ZnCl₂ on carbonization of cellulose when 260 wt % of activating agent was added (61.8% ZnCl₂). In the absence of zinc chloride, the decomposition of cellulose occurs at about 360 °C. When ZnCl₂ is added, the initial dehydration peaks are shifted to temperatures below 250 °C, and a pronounced peak representing decomposition of zinc chloride is present at 600 °C. The first two peaks on the DTG curves in nitrogen (33% weight loss) are accompanied by an endothermic effect seen on the DTA curves. These peaks correspond to the carbonization of cellulose in the presence of ZnCl₂. The weight loss represented by the third peak is smaller than the amount of ZnCl₂ added, which suggests that changes in the organic phase involving incorporation of zinc chloride also occur during carbonization.

That acid washing affects the chemistry of these materials can be seen in the DTG curves in nitrogen presented in Figure 2. For the initial samples pyrolyzed at temperatures below 800 °C, peaks representing removal of zinc chloride (between 500 and 700 °C) and decomposition of other inorganic matter (around 900 °C) are present. For the samples treated at higher temperatures, only the latter peak is seen. After acid washing, those peaks disappeared, indicating the removal of a significant amount of an inorganic material. These species decomposed during pyrolysis at the higher temperatures. The curves for the low-temperature-treated samples, SCZn-1A and SCZn-2A, have more pronounced features than those for SCZn-3A and SCZn-4A. This suggests that heating above 800 °C stabilizes inorganic species and probably makes them less susceptible to acid treatment, as shown in Table 1.²⁴ The peak at 250 °C for SCZn-1A and SCZn-2A is probably the result of the conversion of sulfur species initially present in the Terrene to sulfuric acid during acid treatment and addition of hydrogen peroxide. This peak is much smaller in the case of the samples carbonized at high temperatures, as most of the sulfur was removed during pyrolysis. The initial sludge contains 0.7% sulfur. If some part of this sulfur is present in the form of thermally stable pyrites (FeS₂), it will not be decomposed when heated to 1000 °C. During acid washing,

HCl reacts with sulfur salts, resulting in the formation of H₂S, which is further oxidized by oxygen or H₂O₂ to SO₂ (the odor of H₂S was detected during HCl treatment).

The peak that appears at around 900 °C on DTG curves for samples carbonized at 950 °C can be related to the decomposition of carbonates or the removal of chemically bound oxygen. After pyrolysis, the material is very reactive, and storage in the atmosphere might slightly change the surface properties.

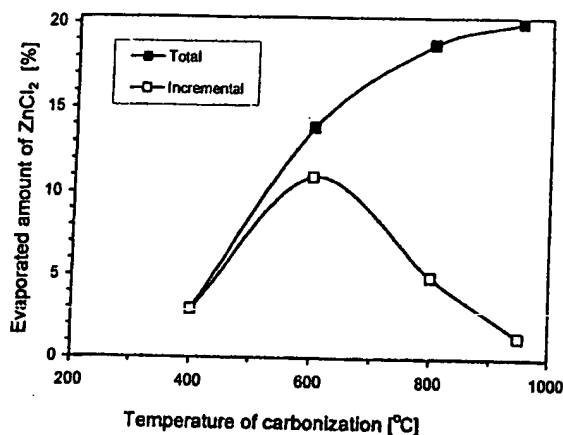
The porous structure parameters calculated from the nitrogen adsorption isotherms using the BET and DFT^{17,18} methods are given in Table 2. These data indicate that, indeed, surface area and pore volume were increased by a factor of 4 for the sample pyrolyzed at 800 °C compared to the lower-temperature-treated materials. This supports our hypothesis concerning the critical role of volatilized zinc chloride molecules in the process of pore formation. The increase in microporosity, as well as in mesoporosity, suggests changes in the composition of the inorganic phase. As shown elsewhere,³ for the Terrene-derived materials, mesoporosity has its origin in the nature of the inorganic (oxide) phase.

After acid washing, the pore structure parameters increased significantly for all samples.⁴ The largest increase was found for the SCZn-2A sample, whose surface area and pore volume increased more than 5-fold. When the SCZn-2 sample was subjected to water washing, a 3-fold increase in those parameters was observed. Such a significant effect of acid washing is related to the removal of excess ZnCl₂,²¹ along with chlorides of other metals such as iron, copper, or nickel.⁴ Water washing was effective only for the dissolution of excess zinc chloride and small quantities of other soluble salts. When carbonization was performed at temperatures higher than 600 °C, the effect of water washing was not as pronounced because of the changes in the chemical nature of the inorganic species and evaporation of zinc chloride during carbonization. It is likely that spinel-like forms of zinc and other metals such as copper or nickel are formed.²³ Because the SCZn-3A and SCZn-4A samples do not react easily with hydrochloric acid, their surface areas and pore volumes increased by less than 50%. The relative microporosity, expressed as the ratio of the volume of micropores to the total pore

Table 3. Weight Loss on Acid Washing, Polarographic Analysis of Zn Content, and Change in Micropore Volume with Pyrolysis Temperature

sample	weight loss (%)	Zn ²⁺ removed (mg/g)	ZnCl ₂ removed (%)	ZnCl ₂ evaporated (%)	volume of ZnCl ₂ evaporated ^a (cm ³ /g)	$\Delta V_{\text{mic}}(\text{DFT})^b$ (cm ³ /g)
SCZn-1	51.8	99.2	20.7	2.9	0.010	—
SCZn-2	38.5	47.0	9.8	13.8	0.047	0.016
SCZn-3	34.7	23.5	4.9	18.7	0.064	0.067
SCZn-4	26.9	17.3	3.6	20.0	0.069	0.069

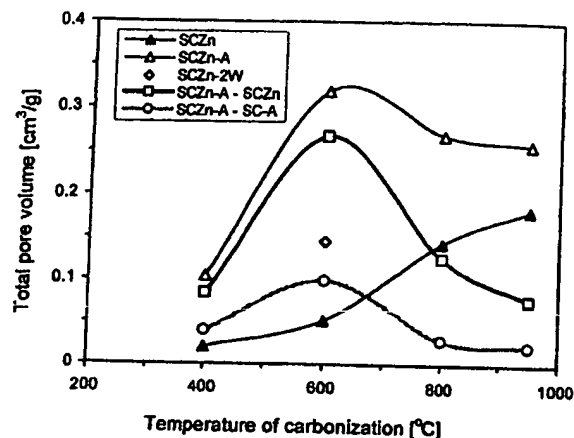
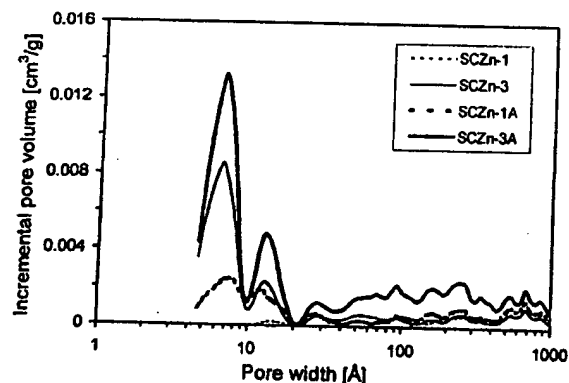
^a Initial ZnCl₂ content in impregnated Terrene was 23.6%. ^b $\Delta V_{\text{mic}} = V_{\text{mic}}(T) - V_{\text{mic}}(400)$.

**Figure 3.** Changes in total and incremental amount of evaporated ZnCl₂ for materials obtained at various temperatures.

volume, is higher for the initial samples than for their acid-treated counterparts. The hydrochloric acid removed inorganic matter from all accessible surfaces, significantly increasing the total pore volume.

The effect of acid washing on zinc chloride removal is presented in Table 3 and Figure 3. For sample SCZn-1, which was carbonized at 400 °C, approximately 21% of the weight loss is associated with dissolution of zinc chloride. On the other hand, for SCZn-3 and SCZn-4, acid washing accounts for only approximately 4% of the weight loss. These results clearly demonstrate the high-temperature volatilization of most of the zinc chloride. A comparison in Table 3 of the volumes of ZnCl₂ evaporated (calculated using its crystalline density) and the increased volume of micropores for the high-temperature samples shows remarkably similar values, especially for SCZn-3 and SCZn-4. Again, this clearly demonstrates the predominant contribution of zinc chloride evaporation to the development of microporosity.

Variations in the zinc chloride content of our samples as a function of carbonization temperature are shown in Figure 3. The incremental trend again demonstrates that 600 °C is the critical temperature when the changes in the pore volumes occur. Changes in the pore volume with increasing carbonization temperature for the Terrene-derived adsorbents with and without washing are presented in Figure 4. The plateau at temperatures higher than 800 °C suggests stabilization of the chemical composition of the adsorbent after evaporation of the zinc chloride. To visualize the effect of zinc chloride removal by acid washing on the development of porosity, the total pore volume without acid washing was subtracted from the volume obtained after acid washing. The curve shows a well-defined maximum at 600 °C that corresponds to the maximum obtained in the incremental zinc chloride removal change (Figure 3). Such a maximum is also seen on the curve obtained by subtrac-

**Figure 4.** Total pore volume as a function of the temperature of carbonization and effect of ZnCl₂ removal by evaporation or washing on the development of pore volume.**Figure 5.** Pore size distributions for selected SCZn samples.

tion of the pore volume obtained after acid washing for the samples carbonized without zinc chloride from that of their counterparts carbonized with zinc chloride.

This stabilization of the pore structure for samples carbonized at temperatures higher than the evaporation temperature of zinc chloride is also seen in the pore size distributions (Figures 5 and 6). In terms of the increase in microporosity (pores < 20 Å), the difference between SCZn-1 and SCZn-2 is far more pronounced than that between SCZn-3 and SCZn-4. After acid washing, the porosity changes, as shown for SCZn-2A. The shape of the distribution curve for pores smaller than 10 Å suggests a notable contribution of small micropores to the total porosity of this material.

Changes in the chemistry and porosity of the adsorbents obtained from biosolids should influence their capacity to remove hydrogen sulfide from moist air.²⁵⁻²⁹ Figure 7 shows the H₂S breakthrough capacity curves for the samples before and after acid washing. The values of the breakthrough capacities and the pHs of

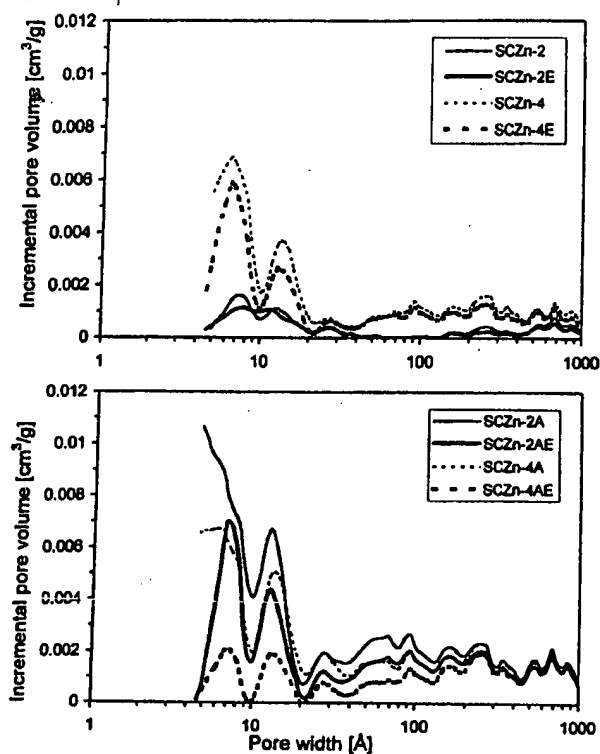


Figure 6. Pore size distributions for the Terrene-derived samples before and after H_2S adsorption.

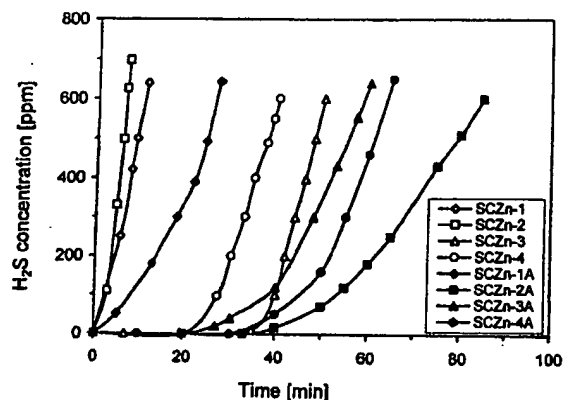


Figure 7. H_2S breakthrough curves for initial Terrene-derived adsorbents before and after acid treatment.

the exhausted materials are reported in Table 1. The capacities of the starting materials are smaller than the capacities of adsorbents obtained by pyrolysis of Terrene, as described elsewhere.⁴ The performance is enhanced by heating to 800 °C, which is probably related to the development of porosity. Also, after acid washing, the capacity increases. This enhancement is more pronounced for the low-temperature-treated samples. This trend follows that revealed in the development of pore structure.

Figure 8 shows the dependence of the breakthrough capacity on the surface area of the adsorbents. Results characterizing the performance of materials obtained from the pyrolysis of Terrene, which are described elsewhere,⁴ are also included. It is clearly seen that, for products of pyrolysis with zinc chloride, the capacity increases with surface area. The correlation coefficient obtained using a linear fit is equal to 0.90. It has to be

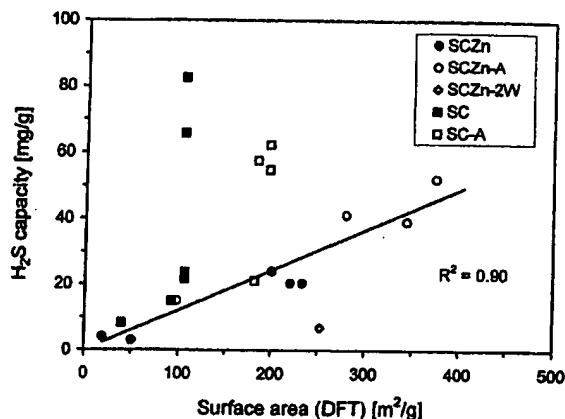


Figure 8. Dependence of the H_2S breakthrough capacity on the surface areas of the adsorbents.

mentioned that, for this fit, we did not take into account the data for SC, SC-A, and SCZn-2W. For SC and SC-A,^{3,4} much higher capacities were obtained, despite the much lower surface areas resulting from the catalytic effect of mixed metal oxide species created during pyrolysis at 950 °C.^{4,24} The existence of the apparent trend in the relationship between breakthrough capacity and surface area for zinc chloride-activated samples (similar specific capacity in milligrams per square meter) and the lack of a trend for SC samples suggests different mechanisms of H_2S removal for these materials.

Adsorbed hydrogen sulfide can be oxidized to sulfuric acid and/or sulfur, or it can react with inorganic components of the adsorbents to form the corresponding sulfides and sulfates.^{4,24-27} As shown elsewhere, for samples obtained by the pyrolysis of Terrene, a significant amount of sulfur in the exhausted materials is in the form of sulfur-containing salts.⁴ Results of the study of hydrogen sulfide adsorption on adsorbents obtained by pyrolysis of Terrene with ZnCl_2 are presented in Figures 9 and 10 and Table 4. The DTG curves in nitrogen for the exhausted, non-acid-washed samples (Figure 9) show two peaks representing weight losses between 200 and 350 °C and between 350 and 500 °C. The first broad peak is assigned to loss of sulfur dioxide,³⁰ and the second to volatilization of elemental sulfur.³¹ The intensity of the first peak increases with an increase in the temperature of carbonization, which is related to increases in the surface area, the pore volume, and the amount of adsorbed hydrogen sulfide.^{14,25-27}

In the case of the exhausted, acid-washed samples, the main weight loss occurred between 200 and 350 °C (Figure 10). The intensity of this peak is related to the amount of hydrogen sulfide adsorbed and oxidized to SO_2 and H_2SO_4 . The peak corresponding to sulfur loss is not present, indicating the excellent selectivity for oxidation of hydrogen sulfide to sulfuric acid. This finding is supported by the very low pH values of the surface after the breakthrough tests.

The weight losses associated with thermal removal of sulfur dioxide and elemental sulfur are presented in Table 4, along with a comparison of the sulfur contents estimated from the breakthrough capacity experiments (S_{BTH}) and thermal analysis (S_{TA}) (the peaks representing SO_2 and S). In the case of non-acid-washed samples, the amounts of sulfur estimated from TA and from the breakthrough capacity measurement do not agree,

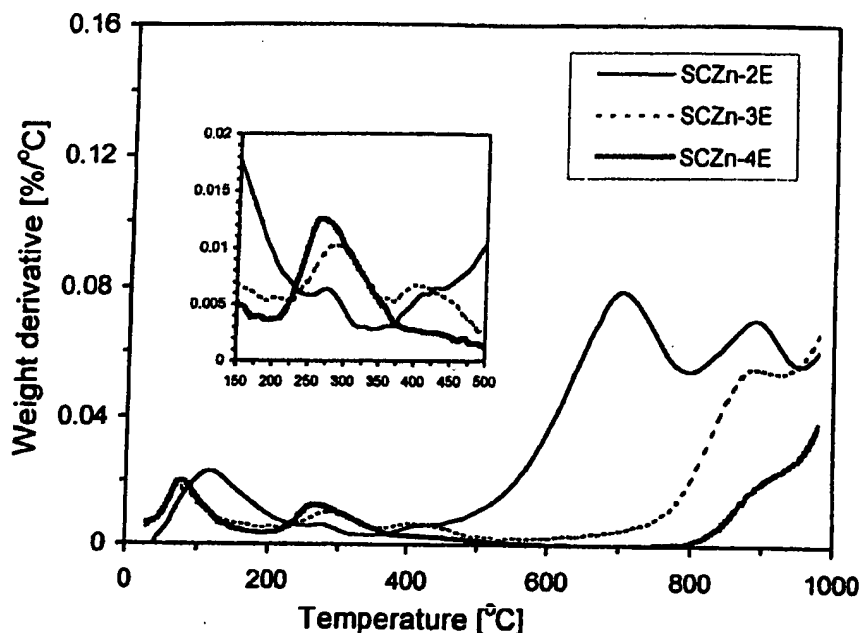
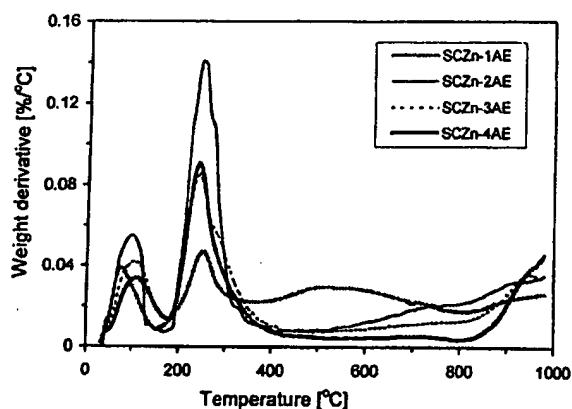


Figure 9. DTG curves in nitrogen for exhausted adsorbents.

Figure 10. DTG curves in nitrogen for acid-treated materials after H₂S adsorption.

indicating formation of sulfides that decompose at temperatures higher than 1000 °C (our maximum temperature).⁴ Another possible reason for the observed discrepancy might be mass-transfer limitations and inhomogeneities in the adsorbent bed after 500 ppm of H₂S was reached. In contrast, for the acid-washed samples good agreement was found. Taking this into account, significant differences exist in the removal process mechanisms caused by differences in surface chemistry and adsorbent porosity. Acid washing removed the metal species capable of reacting with H₂S and produced a microporous surface. Because of the high dispersion of the adsorption sites, H₂S was oxidized to sulfur radicals, as has been shown for microporous activated carbons with basic sites present.^{14,27} These adsorbents, because of the formation of SO₂, show promise from the point of view of the possibility of being regenerated.^{32,33}

The products of sulfur adsorption/oxidation affect the pore size distributions presented in Figure 6. For all samples, a decrease in the volume of small pores is noticed, especially for pores smaller than 10 Å, as has been observed previously for activated carbons.^{14,25-27} The calculated values of the decrease in the volume of

Table 4. Weight Loss (%) in the Temperature Ranges Related to the Presence of the Products of H₂S Oxidation and Estimated Amount of Sulfur from Breakthrough Capacity Test (*S_{BTH}*) and Thermal Analysis (*S_{TA}*)

sample	150–350 °C	350–500 °C	500–700 °C	<i>S_{TA}</i>	<i>S_{BTH}</i>
SCZn-1	1.36	4.15	19.05		
SCZn-1E	—	—	—	NA	0.38
SCZn-2	1.16	0.74	8.31		
SCZn-2E	1.32	1.06	7.10	0.40	0.28
SCZn-3	0.77	0.28	0.16		
SCZn-3E	1.46	0.78	0.53	0.85	2.26
SCZn-4	0.35	0.11	0.00		
SCZn-4E	1.48	0.38	0.02	0.84	1.92
SCZn-1A	4.41	3.67	5.15		
SCZn-1AE	5.26	3.74	5.47	0.50	1.41
SCZn-2A	3.12	1.17	2.36		
SCZn-2AE	11.23	1.55	2.48	4.44	4.89
SCZn-3A	1.80	0.90	1.29		
SCZn-3AE	8.54	1.51	1.82	3.98	3.67
SCZn-4A	1.94	0.67	0.70		
SCZn-4AE	8.07	1.01	0.94	3.41	3.86
SCZn-2W	1.59	1.31	2.13		
SCZn-2WE	1.59	2.05	2.70	0.74	0.65

pores after H₂S removal are presented in Table 2. Assuming that the density of the oxidation products (sulfur or H₂SO₄) is 2 g/cm³, an estimated volume of the deposited products was calculated and reported as *V_{sulf}*. The results suggest that, in the case of the Terrene-derived adsorbents obtained by pyrolysis with zinc chloride, the decrease in the volume of micropores corresponds to the estimated volume of oxidation products. It follows that the micropores are gradually filled with the sulfur products as the process proceeds. Taking into account the fact that, for these materials, the amount of sulfur adsorbed and detected using the TA method was not in agreement with that determined by the breakthrough method, this finding supports our hypothesis that a significant amount of sulfur is bound to the surface in the form of sulfides or sulfates. The surface should then be covered gradually.

The results obtained for the acid-washed samples suggest a different mechanism. In this case, the estimated volume of deposited sulfur is much smaller than the decrease in the volume of micropores or the total

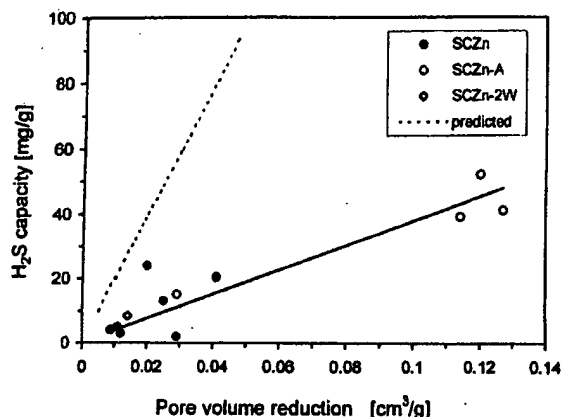


Figure 11. Dependence of the H₂S breakthrough capacity on the pore volume occupied by the oxidation products.

pore volume. It is likely that, for these materials, as for some microporous activated carbons, sulfur products are adsorbed at the entrances of pores, blocking access to other molecules.^{4,34}

The dependence of the H₂S breakthrough capacity on the pore volume reduction is plotted in Figure 11. If deposition of reaction products occurs gradually in all pore space of the adsorbents, then the pore volume reduction should correspond to the capacity of the carbons, expressed in cubic centimeters of products (sulfur or sulfuric acid) per gram of carbon bed. This case is shown as the "predicted" line. For samples studied in this experiment, a linear relationship is noticed between H₂S capacity and pore volume reduction because of the deposition of oxidation products. The slope of the predicted line is greater than that obtained in our experiments, suggesting that not all of the pore volume is used and that adsorbents are exhausted when only a fraction of the available pore volume is occupied. This could result either from the exhaustion of catalytic centers or the blocking of pore entrances by oxidation products.

Conclusions

High-temperature pyrolysis of Terrene with zinc chloride leads to the production of porous materials. Washing with acid causes the removal of a significant quantity of inorganic species, thereby increasing the microporosity and, as a consequence, the hydrogen sulfide breakthrough capacity, which is governed mainly by the adsorbent porosity. On the surface of the materials obtained, hydrogen sulfide is either predominantly bound to active metals to form sulfides and sulfates or oxidized to sulfuric acid. The latter is formed when the content of inorganic matter is reduced after acid washing resulting, in an increase in the porosity of the carbonaceous deposit.

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CURRICULUM VITAE

TERESA J. BANDOSZ, Ph.D.

Assistant Professor
Department of Chemistry
The City College
of The City University of New York
New York, NY 10031

Phone: 212-650-6017

FAX: 212-650-6107

E-mail: tbandosz@scisun.sci.ccny.cuny.edu

Internet: www.sci.ccny.cuny.edu/~tbandosz

EDUCATION

B.Sc., M.Sc., Chemical Engineering, Sept. 1979 - Sept. 1984
University of Mining and Metallurgy,
Department of Processing of Coal in Power Engineering Chemistry
Krakow, Poland

Ph.D., Chemical Engineering, Jan. 1987 - June 1989
Technical University of Cracow,
Department of Chemical Engineering and Technology
Krakow, Poland

Post doctoral Jan. 1991 - July 1996
Syracuse University,
Department of Chemical Engineering and Materials Science

D.Sc., Physical/Analytical Chemistry June 1998
Department of Chemistry
M. Curie-Sklodowska University
Lublin, Poland

ACADEMIC EXPERIENCE

<u>Associate Professor</u>	<u>Jan 2002- Present</u>
<u>Assistant Professor</u>	<u>Sept. 1997- 2000</u>
Department of Chemistry	
The City College of New York	
New York, New York 10031	

Research on sorption of odorous pollutants on activated carbons, preparation of new catalysts for filamentous carbon growing, study of surface chemistry and structure of activated carbons and clay minerals. Desulfurization of air and fuel gas.

Grant Assistant Professor
 Department of Chemistry
 The City College of New York
 New York, New York 10031

Aug. 1996-Aug. 1997

Post doctoral research associate
 Syracuse University,
 Department of Chemical Engineering
 and Materials Science
 Syracuse, NY

Jan. 1991 - 1996

Research work in Hydrogen Storage Program; modification and characterization of activated carbons used for hydrogen storage. Methods applied: static and dynamic (chromatographic) adsorption, X-ray diffraction, mass titration, Boehm titration, potentiometric titration. Evaluation of pore structure and surface chemical properties. Study of clay minerals as matrix for activated carbon produced from polymers. Study of carbons by Small-Angle X-Ray Scattering.

Assistant Professor
 University of Mining and Metallurgy,
 Institute of Energochemistry of Coal
 and Physical Chemistry of Sorbents
 Krakow, Poland

Aug. 1984 - Jan. 1991

Research work on clay minerals. Modification of clays by intercalation. Study of the surface and structure properties. Application of modified clays as sorbents and molecular sieves. Analysis by measurement of adsorption isotherms, helium and mercury density, X-ray diffraction, TGA. Teaching experience in general and organic chemistry.

HONORS AND AWARDS

- Award for Scientific Research, The Chancellor of University of Mining and Metallurgy
- Polish Academy of Sciences Award, The Committee on the Physical Chemistry of Surfaces.
- On Scientific Committee of Pacific Basin Conference on Adsorption Science and Technology II, Australia, May 2000.

PROFESSIONAL AFFILIATIONS

- Polish Mineralogical Society

- International Adsorption Society
- American Institute of Chemical Engineers
- American Chemical Society
- American Carbon Society

RESEARCH AWARDS/FELLOWSHIPS

- National Research Council/ National Academy of Sciences Fall 1997
Travel grant to Ukraine to start collaboration with Ukrainian Academy of Sciences
- NYC DEP Jan. 98-Jan. 99
Study of Granular Activated Carbons \$272,319
- PSC-CUNY July 98- June 00
Effect of Surface Chemistry of Activated Carbons on Sorption of Polar Molecules \$7,910
- NASA (PAIR)(associate investigator) June 98-June 01
Remote Sensing and Environmental/Climate Research
- NYC DEP May 99-May 02
Study of Granular Activated Carbons \$682,896
Travel budget (\$ 1,000.00 /year).
- CUNY Collaborative Incentive Grants program Sept.99-Aug.01
Activated Carbons Obtained from New York City Municipal Sludge as Sorbents for Sulfur Dioxide \$32,000
- Graduate Research Technology Initiative 1999
Evaluation of the levels of Contaminants and Pollutants in Water and Air in New York \$50,000
- PRF Sept.00-Aug.04
The Mechanism of Methyl Mercaptan Adsorption/Oxidation on Activated Carbons \$60,000
- E.I. de Pont de Nemours and Company Feb.01-Jan.02
Determination of Surface pK_a Distributions using Titration Methods in Polar and Non Polar Solvents \$ 15,000
- Synagro Corporation Oct. 01-Sept. 02
Terrene® Modified with Spent Oil as a Precursor for Efficient Adsorbents for H_2S and SO_2 removal. \$ 11,000
- NYC DEP May 02-May 05
Study of Granular Activated Carbons \$476,490
- NATO (Collaborative Linkage Grant: USA-Spain-Ukraine) April01-May 04
Adsorption of hydrogen sulfide on Nitrogen Modified Activated carbons \$ 25,000
- CRDF (USA- Moldava): March02-Apr.04
Activated Carbons for Water Treatment \$5,800
- NYSERDA Feb 04-June04
Sewage Sludge enriched adsorbents Removal of Acidic Gases \$41,000

PUBLICATIONS

- 136 Journal and other Refereed Articles
- 74 Presentations at International Conferences in the U. S., Europe and Asia
- 3 U.S.Patents

(Publications follow on pages 4-19)

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78. Desulfurization From Gaseous Phase On Carbonaceous Adsorbents- Plenary Talk

Teresa J. Bandosz

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